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. ATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

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08 November 1999 (08.11.99)	09 November 1998 (09.11.98)

Applicant

DANIELSSEN, Tore et al

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2.	The election X was was was not
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INTERNATIONAL PRELIMINARY EXAMINATION REPORTS 0 27 FEB 2001

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Applicant's or agent's file reference	FOR FURTHER ACT	ION See Notif	ication of Transmittal of International Examination Report (Form PCT/IPEA/416)
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International application No.	International filing date (day/month/year)	Priority date (day/month/year)
PCT/NO99/00336	08.11.1999		09.11.1998
International Patent Classification (IPC) o	r national classification an	d IPC7	
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Applicant			
Elkem ASA et al			
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This report contains indications re	elating to the following iter	ns:	
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I Basis of the report			
II Priority			
III Non-establishment o	f opinion with regard to no	velty, inventive step	and industrial applicability
IV Lack of unity of inve	ention		
		gard to novelty, inve	ntive step or industrial applicability; citations
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VI Certain documents of			
VII Certain defects in the	e international application		
VIII Certain observations	on the international applic	ation	
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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/NO99/00336

I. Basis of th	ne report		•
1. This report l	has been drawn or 14 are referred to in	n the basis of (Replacement shi this report as "originally filed	eets which have been furnished to the receiving Office in response to an invitation "and are not annexed to the report since they do not contain amendments.):
\boxtimes	the international	application as originally file	ed.
	the description,	pages	, as originally filed,
			_, filed with the demand,
			_ , filed with the letter of ,
		pages	_ , filed with the letter of
	the claims,	Nos.	_ , as originally filed,
		Nos.	_ , as amended under Article 19,
		Nos.	_ , filed with the demand,
			_ , filed with the letter of ,
		Nos.	_ , filed with the letter of
	the drawings,	sheets/fig	_ , as originally filed,
		sheets/fig	_ , filed with the demand
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beyo	and the disclosure	as filed, as indicated in the	supplemental Box (Rule 70.2(c)).
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4. Additional	observations, if in	eccssary.	



INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/NO99/00336

Resoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

 Novelty (N)
 Claims
 1-7
 YES

 Claims
 NO

 Inventive step (IS)
 Claims
 1-7
 YES

 Claims
 NO

 Industrial applicability (IA)
 Claims
 1-7
 YES

 Claims
 NO

2. Citations and explanations

The claimed invention relates to thermoplastic resin compositions, a method for the production of the compositions and a filler blend for use in the compositions.

Talc is used in polyolefines such as polypropylene to increase the stiffness of the polypropylene product. In combination with filler in order to improve for example impact strength it has been found that the stiffness is substantially reduced. To solve this problem it is therefore an object of the present invention to provide thermoplastic resins having both high stiffness and high impact strength.

According to the invention, the solution is to use a combination of talc and microsilica as fillers in thermoplastic resins, which gives a product having both high stiffness and high impact strength.

Cited documents in the International Search Report:

D1 US 4956404 A
D2 US 5266609 A
D3 US 4714733 A
D4 US 4140669 A
D5 US 4722952 A

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International application No.

PCT/NO99/00336

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

Documents D1 to D4 disclose the use of talc and silica in discloses а thermoplastic D1 thermoplastic compositions. composition containing talc and fumed silica with the quantity and the weight ratio between talc and silica within the values of the claimed invention, see example 1, column 5 and 6. In D4, a thermoplastic composition containing talc and silica is disclosed. The silica component can have a particle size of less than 325 mesh, see column 3, line 22. However, the fumed silica in D1 has much smaller particle size and in D4 the silica used has higher particle size than microsilica in the present application. Thus, the invention claimed is novel.

The claimed invention differs from the subject matter in documents D2 and D3. In those documents talc is only mentioned as one example of fillers among several. D5 discloses a thermoplastic composition containing silica fume. A conduit of PVC with fume silica shows improved impact resistance but D5 does not mention any use of talc.

Besides, none of the documents says anything of the problem above. Therefore, it is not considered obvious to a person skilled in the art to arrive at the claimed invention with the knowledge from the cited documents.

In accordance with the arguments above the invention claimed in claims 1-7 is novel, is considered to involve an inventive step and to have industrial applicability.



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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(71) Applicants (for all designated States except US): ELKEM ASA [NO/NO]; Hoffsveien 65B, N-0377 Oslo (NO). MONDO MINERALS OY [FI/FI]; Laurinmäenkuja 3B, FIN-00440 Helsinki (FI).

(72) Inventors; and

- (75) Inventors/Applicants (for US only): DANIELSSEN, Tore [NO/NO]; Åsveien 20, N-4621 Kristiansand (NO). LIN-NEBO, Anne, Kathrine [NO/NO]; Nedre Frydendal 106, N-1370 Asker (NO). SANDELIN, Bjørn [FI/FI]; Käenpolku 3, FIN-87400 Kajaani (FI).
- (74) Agent: VINDENES, Magne; Elkem ASA Patent Dept., P.O. Box 8040 Vågsbygd, N-4675 Kristiansand (NO).

(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

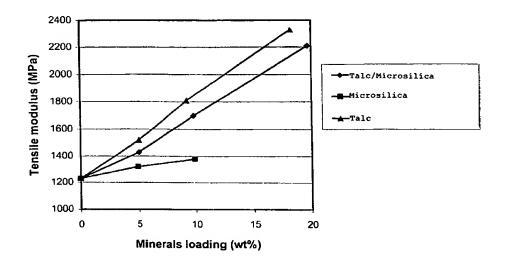
Published

With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: RESIN COMPOSITIONS, METHOD OF PRODUCING RESIN COMPOSITIONS AND FILLER BLENDS FOR USE IN RESIN COMPOSITIONS

Stiffness of talc/EMS in PP copolymer



(57) Abstract

The present invention relates to thermoplastic resin compositions, particularly polyolefines, polyvinylchloride and polyamide. The thermoplastic resin compositions contain between 3 and 400 % by weight of filler based on the weight of the resin, said filler comprising talc and microsilica where the weight ratio between talc and microsilica is between 15:1 and 1:15. The invention further relates to a method for the production of thermoplastic resin compositions, and to a filler blend for use in thermoplastic resins, said blend containing talc and microsilica in a weight ratio between 15:1 and 1:15.

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WO 00/27911 PCT/NO99/00336

Title of Invention

Resin compositions, method of producing resin compositions and filler blends for use in resin compositions.

5 Technical Field

The present invention relates to new and improved resin compositions and more particularly to thermoplastic resin compositions such as polyolefines, polyvinylchloride and polyamide, and to a method for the production of resin compositions. The invention further relates to a filler blend for use in the production of resin compositions.

Background Art

It is well known to produce polyolefines such as polypropylene compound containing functional fillers such as fine particulate talc to increase the stiffness of the final polypropylene product.

Talc is hydrated magnesium silicate with the theoretical formula 3MgO.4SiO₄.H₂O and consists of magnesiumhydroxide sandwiched between two sheets of silica.

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When adding other fillers in addition to talc in order to improve other properties, such as for example impact strength, it has, however, been found that the stiffness obtained by using talc alone as a filler is substantially reduced when adding a second filler for increasing the impact strength. It has therefore not been possible to produce polypropylene products with both a high stiffness and a high impact strength. High stiffness and high impact strength is particularly important in some polypropylene products such as for example car bumpers. The same is true for other thermoplastic resin products.

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The term thermoplastic resin used in the specification and claims includes not only thermoplastic resins per se, but also mixtures thereof, as well as a blend

of thermoplastic resins with other materials such as an elastomer like nitrile rubber. The so-called thermoplastic rubbers, thermoplastic elastomers are also included in the definition of thermoplastic resin. Thermoplastic resins per se includes polyolefines, polystyrene, polyesters, ABS copylymers, polyvinyl chloride (PVC), unplasticized polyvinyl chloride (UPVC), polyamide, acrylic polymers, polycarbonate polymers, polysulfone polymers and others.

It is known from US patent No. 4,722,952 that the addition of microsilica to polyvinylchloride, improves the impact strength of polyvinylchloride used for the production of electrical conduits. For such products the stiffness is of no importance. On the contrary, high stiffness is not desired for electrical conduits.

The term microsilica used in the specification and claims is particulate amorphous SiO₂ obtained from a process in which silica is reduced and the reduction product is oxidized in vapor phase to form amorphous silica. Microsilica may contain at least 70 % by weight silica (SiO₂) and have a specific density of 2,1 - 2.3 g/cm³ and a surface area at 15 - 30 m²/g. The primary particles are substantially spherical. The primary particles have an average size of about 0,15µm. Microsilica is preferably obtained as a coproduct in the production of silicon or silicon alloys in electric reduction furnaces. In these processes large quantities of silica are formed as SiO₂. The SiO₂ is recovered in conventional manner using filter or other collection apparatus. For the purpose of the present invention the term microsilica also shall be understood to include fly-ash, and more particularly fly-ash particles of substantial spherical shape having a particle size below 10 microns.

Disclosure of Invention

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It is an object of the present invention to provide thermoplastic resins having both high stiffness and high impact strength.

According to a first aspect, the present invention thus relates to thermoplastic resin compositions, particularly polyolefines, polyvinylchloride and polyamide, characterized in that the thermoplastic resin compositions contains between 3 and 400 % by weight of filler based on the weight of the resin, said filler comprising talc and microsilica where the weight ratio between talc and microsilica is between 15:1 and 1:15.

According to a preferred embodiment the weight ratio of talc and microsilica is between 6:1 and 1:5.

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According to a second aspect the present invention relates to a method for the production of thermoplastic resin composition, particularly polyolefines, polyvinylchloride and polyamide, which method being characterized in that talc and microsilica is added to thermoplastic resin in a total amount between 3 and 400 % by weight based on the weight of thermoplastic resin and where the weight ratio between talc and microsilica is kept between 15:1 and 1:15, whereafter the mixture is formed into a thermoplastic resin product or compound.

According to a preferred embodiment of the method of the present invention talc and microsilica are added to the thermoplastic resin as a mixture of talc and microsilica.

The compounding of the termoplastic resin can be done using conventional processes like extrusion, calendering, injection moulding and others.

According to a third aspect, the present invention relates to a filler blend for use in thermoplastic resins, particularly polyolefines, polyvinylchloride and polyamide, wherein the filler blend contains talc and microsilica in a weight ratio between 15:1 and 1:15, and particularly between 6:1 and 1:5.

It has surprisingly been found that the combined use of talc and microsilica as fillers in thermoplastic resins, particularly in polyolefines, polyvinylchloride and polyamide, give final products having both high stiffness and high impact strength.

EXAMPLE 1

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A non-filled polypropylene copolymer "BA 202E" supplied by Borealis was extruded in a compounding extruder with addition of a filler blend consisting of talc supplied by Mondo Minerals OY and microsilica supplied by Elkem ASA. The weight ratio between talc and microsilica in the filler blend was 2:1 and tests were run with addition of 5,10 and 19 % by weight of the filler blend based on the weight of the polypropylene copolymer. The stiffness of the extruded polypropylene was measured as tensile modulus according to ISO 527 and the impact strength of the extruded polypropylene was measured as notched charpy impact strength according to ISO 179/1A.

For comparison purposes the polypropylene copolymer was extruded in the compounding extruder with no addition of filler and with the addition 5, 10 and 18 % by weight of talc and with 5 and 10 % by weight of microsilica. Also for these comparative tests the stiffness and the impact strength were measured as stated above. The resulting stiffness and impact strength are shown in figure 1 and figure 2 respectively.

As can be seen from figure 1 and 2, the impact strength of the polypropylene containing both talc and microsilica is much higher than for the polypropylene containing only talc and only slightly lower than for the polypropylene containing only microsilica as a filler. The stiffness of the polypropylene containing both talc and microsilica is much higher than for polypropylene containing only microsilica as a filler and only slightly lower than for polypropylene containing only talc as a filler. The use of a blend of talc and

microsilica thus surprisingly gives a polypropylene having both a high stiffness and a high impact strength.

EXAMPLE 2

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A non-filled high density polyethylene (HDPE) copolymer "HDPE HE 2467-BL" supplied by Borealis was extruded in a compounding extruder with additon of a filler blend consisting of talc supplied by Mondo Minerals OY and microsilica supplied by Elkem ASA. The weight ratio between talc and microsilica in the filler blend was 2:1 and the test was run with addition of 10 % by weight of the filler blend based on the weight of the HDPE copolymer. The stiffness of the extruded HDPE was measured as tensile modulus according to ISO 527 and the impact strength of the extruded HDPE was measured as notched charpy impact strength according to ISO 179/1A.

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For comparison purposes the HDPE copolymer was extruded in the compounding extruder with no addition of filler, with the addition 10 % by weight of talc and with addition of 10 % by weight of microsilica. Also for these comparative tests the stiffness and the impact strength were measured as stated above. The resulting stiffness and impact strength are shown in table 1.

Table 1

	Tensile	Impact Strength
	Modulus	(kJ/m²)
Material	(MPa)	
HDPE nonfilled	850	13.6
HDPE + 10 % talc	1160	18.0
HDPE + 10 % microsilica	880	27.6
HDPE + 10 % filler blend	1070	22.3

As can be seen from table 1, the impact strength of the HDPE containing both talc and microsilica is higher than for the HDPE containing only talc, but lower than for the HDPE containing only microsilica as a filler. The stiffness of the HDPE containing both talc and microsilica is much higher than for HDPE containing only microsilica as a filler and only slightly lower than for HDPE containing only talc as a filler. The use of a blend of talc and microsilica thus surprisingly resulting in a HDPE having both a high stiffness and a high impact strength.

10 **EXAMPLE 3**

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A non-filled polyvinylchloride (PVC) polymer was calendered with additon of a filler blend consisting of talc supplied by Mondo Minerals OY and microsilica supplied by Elkem ASA. The weight ratio between talc and microsilica in the filler blend was 2:1 in one run and 1:2 in another run, and the tests were run with addition of 5 % by weight of the filler blend based on the weight of PVC polymer. The stiffness of the calendered PVC was measured as tensile modulus according to ISO 527 and the impact strength of the calendered PVC was measured as notched charpy impact strength according to ISO 179/1A.

For comparison purposes the PVC polymer was calendered with no addition of filler, with addition of 5 % by weight of talc and with addition of 5 % by weight of microsilica. Also for these comparative tests the stiffness and the impact strength were measured as stated above. The resulting stiffness and impact strength are shown in table 2.

Table 2

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Material	Tensile Modulus (MPa)	Impact Strength (kJ/m ²)
PVC nonfilled	2916	6.5
PVC + 5 % talc	3484	5.4
PVC + 5 % microsilica	3010	8.5
PVC + 5 % filler blend talc/microsilica 2:1	3360	5.1
PVC + 5 % filler blend talc/microsilica 1:2	3167	7.9

As can be seen from table 2, the impact strength of PVC containing talc and microsilica in a ratio of 2:1 is about the same as for the PVC containing only talc, but lower than for PVC containing only microsilica as a filler. For PVC containing talc and microsilica in a ratio of 1:2 it can be seen that the impact strength is higher than for PVC containing talc and microsilica in a ratio of 2:1 and almost as high as for PVC containing only microsilica. The stiffness of the PVC containing talc and microsilica in a ratio of 2:1 is much higher than for PVC containing only microsilica as a filler and only slightly lower than for PVC containing only talc as a filler. For PVC containing talc and microsilica in a ratio of 1:2 it will be seen that the tensile modulus is still higher than for PVC containing only microsilica. The use of a blend of talc and microsilica thus surprisingly gives a PVC having both a high stiffness and a high impact strength.

EXAMPLE 4

A non filled polyamide (PA) polymer, "PA6 Ultramid B35" delivered by BASF was extruded in a compounding extruder with addition of a filler blend consisting of talc supplied by Mondo Minerals OY and microsilica supplied by

Elkem ASA. The addition of filler blend was 10 % by weight of polymer. The weight ratio between talc and microsilica in the filler blend in a first test was 1:1 and 1:2 in a the second test. The stiffness of the extruded PA was measured as tensile modulus according to ISO 527 and the impact strength of the extruded PA was measured as notched charpy impact strength according to ISA 179/1A.

For comparison purposes the PA copolymer was extruded in the compounding extruder with no addition of filler, with the addition 10 % by weight of talc and with addition of 10 % by weight of microsilica. Also for these comparative tests the stiffness and the impact strength were measured as stated above. The resulting stiffness and impact strength are shown in table 3.

Table 3

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Material	Tensile Modulus (MPa)	Impact Strength (kJ/m²)
PA nonfilled	700	Non-break
PA + 10 % talc	1430	10.6
PA + 10 % microsilica	890	33.2
PA + 10 % filler blend talc/microsilica 1:1	1210	16.3
PA + 10 % filler blend talc/microsilica 1:2	1120	19.7

As can be seen from table 3, the impact strength of the PA containing both talc and microsilica is much higher than for the PA containing only talc, but lower than for the PA containing only microsilica as a filler. It can also be seen that the impact strength increases with increasing amount of microsilica in the filler blend. The stiffness of the PA containing both talc and microsilica is

much higher than for PA containing only microsilica, but the stiffness is slightly reduced when the microsilica content in the filler blend is increased.

CLAIMS

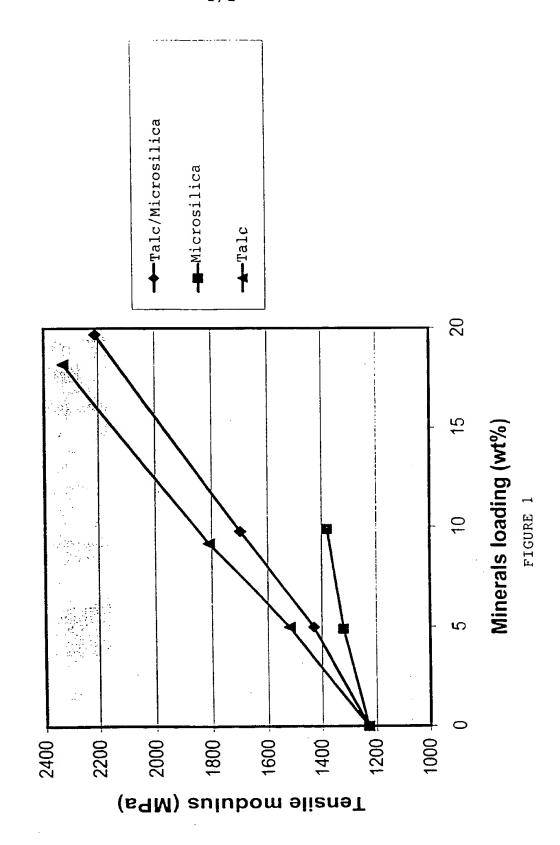
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- 5 1. Thermoplastic resins compositions, particularly polyolefines, characterized polyvinylchloride and polyamide, in that the thermoplastic resin compositions contain between 3 and 400 % by weight of filler based on the weight of the resin, said filler comprising talc and microsilica where the weight ratio between talc and microsilica is between 10 15:1 and 1:15.
 - 2. Thermoplastic resins according to claim 1, characterized in that the weight ratio of talc and microsilica is between 6:1 and 1:5.
- 3. A method for the production of thermoplastic resin composition, particularly polyolefines, polyvinylchloride and polyamide, c h a r a c t e r i z e d i n that talc and microsilica is added to the thermoplastic resin in a total amount between 3 and 400 % by weight based on the weight of thermoplastic resin and where the weight ratio between talc and microsilica is kept between 15:1 and 1:15, whereafter the mixture is formed to a thermoplastic resin product or compound.
 - 4. A method according to claim 3, characterized in that talc and microsilica are added to the thermoplastic resin as a mixture of talc and microsilica.
 - 5. A method according to claim 2, characterized in that talc and microsilica are added separately to the thermoplastic resin.
- 30 6. A filler blend for use in thermoplastic resin compositions, particularly polyolefines, polyvinylchloride and polyamide, characterized in

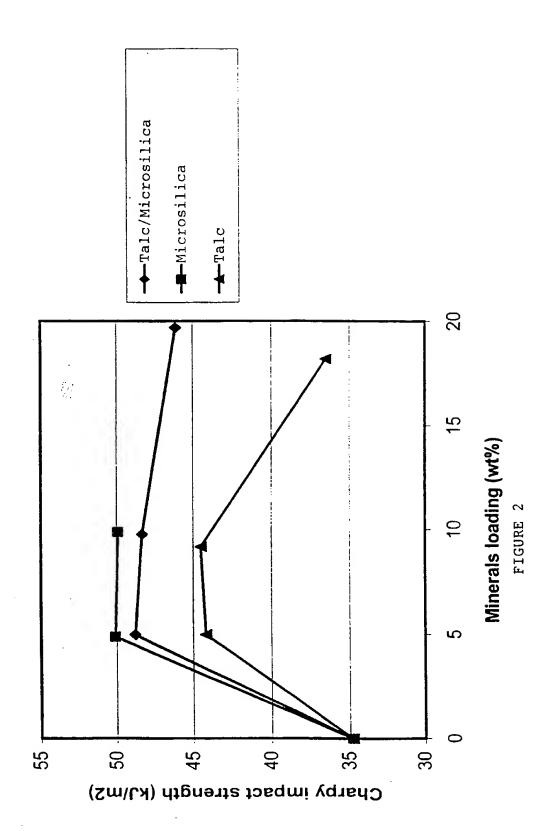
that the filler blend contains talc and microsilica in a weight ratio between 15:1 and 1:15.

7. A filler blend according to claim 6, characterized in that the filler blend contains talc and microsilica in a weight ratio between 6:1 and 1:5.

Stiffness of talc/EMS in PP copolymer



Impact strength of talc/EMS in PP copolymer



INTERNATIONAL SEARCH REPORT

International application No.

PCT/NO 99/00336 A. CLASSIFICATION OF SUBJECT MATTER IPC7: C08K 3/34, C08K 3/36 According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC7: CO8K Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched SE,DK,FI,NO classes as above Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) C. DOCUMENTS CONSIDERED TO BE RELEVANT Category* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. Α US 4956404 A (JOSEF PELZIG), 11 Sept 1990 1-7 (11.09.90), column 4, line 5 - line 14; column 5, line 3 - line 7; column 6, line 1 - line 39, claims 1,12, abstract A US 5266609 A (NEIL R. HALL ET AL), 1-7 30 November 1993 (30.11.93), column 3, line 17 - line 30, claims 1,9 and 12 US 4714733 A (KUNIO ITOH ET AL), 22 December 1987 Α 1-7 (22.12.87), column 6, line 25 - line 38, abstract X Further documents are listed in the continuation of Box C. See patent family annex. Special categories of cited documents: later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "A" document defining the general state of the art which is not considered to be of particular relevance "E" erlier document but published on or after the international filing date "X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other step when the document is taken alone special reason (as specified) document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is document referring to an oral disclosure, use, exhibition or other combined with one or more other such documents, such combination means being obvious to a person skilled in the art document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of mailing of the international search report Date of the actual completion of the international search 19 -04- 2000 13 March 2000 Name and mailing address of the ISA/ Authorized officer Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Barbro Nilsson/Els

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2 INTERNATIONAL SEARCH REPORT

International application No. PCT/NO 99/00336

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